



BSI Standards Publication

**Lubricants - Determination of aerobic
biological degradation of fully formulated
lubricants in an aqueous solution - Test
method based on CO₂-production**

National foreword

This British Standard is the UK implementation of [EN 17181:2019](#).

The UK participation in its preparation was entrusted to Technical Committee MCE/18/-/16, Hydraulic fluids.

A list of organizations represented on this committee can be obtained on request to its secretary.

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English Version

**Lubricants - Determination of aerobic biological
degradation of fully formulated lubricants in an aqueous
solution - Test method based on CO₂-production**

Bio-lubrifiants - Détermination de la
biodégradabilité aérobie, en solution aqueuse,
de lubrifiants complètement formulés -
Méthode basée sur le dégagement de CO₂

Schmierstoffe - Bestimmung der aeroben
biologischen Abbaubarkeit ausformulierter
Schmiermittel in wässrigem Medium -
Testmethode basierend auf CO₂-Bestimmung

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European foreword

This document ([EN 17181:2019](#)) has been prepared by Technical Committee CEN/TC 19 “Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin”, the secretariat of which is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by August 2019, and conflicting national standards shall be withdrawn at the latest by August 2019.

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Introduction

Existing test methods for determination of aerobic biodegradation of organic substances in an aqueous medium (e.g. numerous ISO and OECD test methods) are widely used. Moreover, all actually existing ecolabels, international and national regulations and recommendations are working with them.

Testing of organic substances requires a broad scope of preparation tools to maintain comparable results for all different materials appearing liquid, solid, water-soluble, non-water-soluble or emulsifiable.

In consequence of this, results may differ regarding the choice of test method, preparation procedure or inoculum thus leading to low levels of reproducibility and repeatability.

Fully formulated lubricants (oils or greases or emulsions) are complex mixtures of mostly organic compounds that are mainly water insoluble. Based on [ISO 9439](#) (resp. OECD 301B) this test method has been aligned by modifying sample preparation and test procedure for such complex mixtures in order to achieve improved significance of test results. The test method described in this document, however, was not assessed for greases. The precision statement therefore only applies for lubricating oils.

Finally, from customer's point of view a biodegradation test method for the final product of improved significance will result in better market transparency, enhanced reputation of "bio-lubricants" and confidence of the customer in this product group.

1 Scope

This document specifies a procedure for determining the degree of aerobic degradation of fully formulated lubricants. The organic material in a fully formulated lubricant is exposed in a synthetic aqueous medium under laboratory conditions to an inoculum from activated sludge.

Biodegradation resulting in mineralisation of the organic material can be determined by measuring released CO₂ in a total organic carbon (TOC) analyser. In contrast to existing test methods measuring released CO₂ this method uses a precise preparation procedure for non-water soluble organic material.

The above mentioned method applies to fully formulated lubricants which

- a) are water-soluble, non-water soluble or emulsifiable, and
- b) are not toxic and not inhibitory to the test microorganisms at the test concentration.

The presence of inhibitory effects is determined as specified in this document.

This test method is focused on fresh water as test medium. Tests in sea water are currently not included in this method, but may be introduced later.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

[ISO 9439](#), *Water quality — Evaluation of ultimate aerobic biodegradability of organic compounds in aqueous medium — Carbon dioxide evolution test*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <http://www.iso.org/obp>

3.1

organic material

total amount of all organic compounds a fully formulated lubricant consists of

3.2

aerobic biodegradation

consumption of organic materials by microorganisms in a biochemical process under use of oxygen, resulting in cleavage of chemical bonds and CO₂ production providing energy and/or new biomass

3.3

primary biodegradation

first structural change (transformation) of an organic chemical compound by microorganisms resulting in the change of a specific property

3.4

mineralisation

aerobic biodegradation of organic material by microorganisms to yield carbon dioxide, water and mineral salts of any other elements present and new biomass

3.5

activated sludge

biomass produced in the aerobic treatment of wastewater by the growth of bacteria and other microorganisms in the presence of dissolved oxygen

3.6

concentration of suspended solids of an activated sludge

amount of solids obtained by filtration or centrifugation of a known volume of activated sludge and drying at about 105 °C to constant mass

3.7

inoculum

sample of activated sludge for the purpose of this test method used in degradation procedures described in this method

3.8

reference compound

organic compound of known biodegradability with a degradation degree of more than 60 %

3.9

dissolved organic carbon

DOC

part of the organic carbon in water which cannot be removed by specified phase separation

NOTE For example by centrifugation at 4 000 rpm for 15 min or by membrane filtration using membranes with pores of 0,2 µm to 0,45 µm diameter.

3.10

total inorganic carbon

TIC

total amount of inorganic carbon in a water sample arising from carbon dioxide and carbonates

3.11

dissolved inorganic carbon

DIC

part of the inorganic carbon in water which cannot be removed by specified phase separation

NOTE For example by centrifugation at 4 000 rpm for 15 min or by membrane filtration using membranes with pores of 0,2 µm to 0,45 µm diameter.

3.12

theoretically released amount of carbon dioxide

ThCO₂

theoretical maximum amount of carbon dioxide released from a chemical compound, calculated from TOC content

NOTE Expressed in this case as milligrams of carbon dioxide evolved per milligram or gram of test compound.

3.13

total organic carbon

TOC

amount of carbon bound in an organic compound

NOTE Refer to ISO 8245 [17] for further details.

3.14

biochemical oxygen demand

BOD

mass concentration of dissolved oxygen consumed under specified conditions by the aerobic biological oxidation of a chemical compound or organic matter in water

NOTE Expressed in this case as milligrams oxygen uptake per milligram or gram of test compound.

3.15
chemical oxygen demand
COD

mass concentration of oxygen equivalent to the amount of a specified oxidant consumed by a chemical compound or organic matter when a water sample is treated with that oxidant under defined conditions

NOTE to entry Expressed in this case as milligrams oxygen consumed per milligram or gram of test compound.

3.16
theoretical oxygen demand
ThOD

theoretical maximum amount of oxygen required to oxidize a chemical compound completely, calculated from elemental analysis

NOTE Expressed in this case as milligrams of oxygen uptake per milligram or gram of test compound.

3.17
lag phase

time from the start of a test until adaptation and selection of the degrading microorganisms are achieved and the biodegradation degree of a chemical compound or organic matter has increased to about 10 % of the theoretical maximum biodegradation

NOTE This is expressed in days.

3.18
maximum level of biodegradation

maximum biodegradation degree of a chemical compound in a test, above which no further biodegradation takes place during the test

NOTE This is expressed in percentage.

3.19
biodegradation phase

time from the end of the lag phase of a test until about 90 % of the maximum level of biodegradation has been reached

NOTE This is expressed in days.

3.20
plateau phase

time from the end of the biodegradation phase when the maximum level of biodegradation is reached until the end of the test

3.21
pre-conditioning

pre-incubation of an inoculum under the conditions of the subsequent test in the absence of the test chemical compound or organic matter, with the aim of improving the performance of the test by acclimatization of the microorganisms to the test conditions

3.22
pre-exposure

pre-incubation of an inoculum in the presence of the test chemical compound or organic matter under test, with the aim of enhancing the ability of the inoculum to biodegrade the test material by adaptation and/or selection of the microorganisms

4 Principle

Biodegradation is followed over a specified period by measuring the production of carbon dioxide CO₂. The evolution of carbon dioxide (CO₂) is determined and compared with the theoretical carbon dioxide evolution (ThCO₂) using the CO₂ evolution test as in [ISO 9439](#).